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MEMORANDUM REPORT NO. 2293

ANALYSIS OF EXHAUST GASES FROM THE XM-19
RIFLE -- AN APPLICATION OF GAS
CHROMATOGRAPHY/MASS SPECTROSCOPY

by

J. J. Rocchio
I. W. May

May 1973

"TEST + EVALUATION"

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MEMORANDUM REPORT NO. 2293

MAY 1973

ANALYSIS OF EXHAUST GASES FROM THE XM-19 RIFLE --
AN APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY

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ABERDEEN PROVING GROUND, MARYLAND

BALLISTIC RESEARCH LABORATORIES

MEMORANDUM REPORT NO. 2293

JJRocchio/IWMay/lkg
Aberdeen Proving Ground, Md.
May 1973

ANALYSIS OF EXHAUST GASES FROM THE XM-19 RIFLE--
AN APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY

ABSTRACT

A technique combining gas chromatographic and mass spectrometric analysis was developed and applied to determine the chemical composition of gases resulting from firing the XM-19 rifle with the XM-645 flechette round. Cyanogen, carbonyl sulfide, carbon monoxide, nitrous and nitric oxides were among the products detected. Quantitative data were obtained for some of the components. Thermodynamic equilibrium calculations were performed and compared with the experimental results.

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I. INTRODUCTION

Operator reports of nausea and peculiar odors during a test firing of the XM-19 Rifle and XM-645 flechette round, candidates for the Future Rifle System, gave rise to the concern that the exhaust gas might contain toxic components. The XM-645 contains a propellant (X-2473.13) which, except for granulation, is similar to WC 846 ball propellant. The compositions of the propellant and the piston primer are listed in Appendix A. It also differs from conventional rounds in that it contains a fiberglass sabot and rubber gas seal as shown in Appendix B which could affect the composition of the combustion gas.

At the request of the U.S. Army Small Arms Systems Agency, the Ballistic Chemistry Branch, Interior Ballistics Laboratory (IBL) initiated a project to study the exhaust gas from the XM-19. The objectives of this project were to identify gaseous combustion products which might present a toxicological hazard and to provide quantitative data on these as time and resource limitations imposed at the outset of the project permitted.

An additional objective has been to develop a quick and efficient analytical method using the most up-to-date techniques which can be applied to weapons exhaust analysis in general. A need for this has been demonstrated as a result of the development of modern weapon systems using new materials and designed for high rates of fire and use in relatively confined areas (e.g. a helicopter cabin).

II. BACKGROUND

The evaluation of the toxic hazard of weapon exhaust involves the detection, identification and quantification of the gases in the post-firing environment. This process is made experimentally difficult by the fact that each round produces a small amount of gas (about 1.2% at STP per round) which is rapidly mixed and diluted in the ambient air. Some method of sample concentration coupled with sensitive analytical techniques is required to overcome this difficulty.

Some background information on weapons exhaust analysis is available from Project West¹, a study of the exhaust gases from 7.62mm and caliber 0.50 machine guns in a helicopter environment. Techniques were developed under this task for firing weapons in an enclosed area to inhibit dilution of exhaust products by ambient air. During firing, the exhaust gases were continuously monitored with a gas flow cell and rapid scan infrared spectrometer. Evacuated cylinders and a

* References are listed on page 33

condensation train were used to provide gas samples for subsequent analysis by conventional infrared spectroscopy and high resolution mass spectroscopy. The concentrations of some of the toxic gases found in the 7.62 mm exhaust are listed in Appendix C. In this study, mass spectroscopy proved to be the most versatile method of analysis.

On the basis of this data, it was apparent that the present study would require a technique which was capable of analyzing a complex mixture of components at low concentrations. The technique of combined gas chromatography-mass spectroscopy which is being developed in this laboratory to study a variety of propellant ignition and combustion problems appeared to be ideal for this problem. In this approach a complex mixture is first separated by the gas chromatograph and the components analyzed directly by the mass spectrometer. Identification of the components can be made on the basis of the mass spectrum and the gas chromatographic retention time. Quantitative analysis of the mixture can be performed by conventional gas chromatography.

III. EXPERIMENTAL

A. Mass Spectrometer

The quadrupole mass spectrometer used in these studies was manufactured by Extranuclear Laboratories. The maximum mass range is 0-450 amu. The sweep width is determined by setting the masses at which the sweep is to start and stop. The rate at which this range is swept can be varied from 1 msec to 10 sec per amu. A useful feature is the capability of monitoring the intensity of ions of a single mass as a function of time. The "mass chromatograms" generated in this way facilitate the detection of trace contaminants in complex mixtures. Mass spectra were recorded in analog form on a two-channel Brush 220 recorder.

B. Gas Chromatograph/Mass Spectrometer Interface

Of the many approaches to interfacing a mass spectrometer (MS) to a gas chromatograph, (GC), the direct inlet system was chosen. It is simple and rugged, and eliminates such problems as mass discrimination, membrane selectivity, and absorption effects. The pumping system of the mass spectrometer was designed with a high capacity oil diffusion pump (4000%/sec). The interface consisted of a 1 m length of stainless steel capillary tubing (1.59 mm OD x 0.25 mm ID). This restrictor reduces the pressure from atmospheric at the chromatograph to the working pressure of the mass spectrometer. With this system, up to 20 cc/min of helium carrier gas may be inlet to the mass spectrometer while keeping the pressure in the 10^{-5} torr range. The transfer line is heated to prevent condensation of any of the less volatile components.

C. Gas Chromatograph

A Perkin-Elmer 880 gas chromatograph equipped with a temperature programmer, flame ionization detector (FID) and a Carle micro thermal conductivity detector (TCD) was used throughout these studies. A Hewlett-Packard 3370B electronic integrator was used in the quantitative analyses. Cryogenic analyses at 195°K were performed by packing the sample column in powdered dry ice. The column used for these analyses was a 3m x 3mm OD copper column filled with Porapak QS (80/100 mesh), a commercial porous polymer packing. The analytical equipment is shown in Figure 1.

D. Exhaust Confinement Vessel

The objective of this device was to restrict the diffusion of the exhaust gases into the surrounding air. The confinement vessel was constructed from an aluminum cylinder 1.02 m in length and 0.3 m in diameter. One end was covered with an aluminum plate with an opening to accept the muzzle of the XM-19 rifle. The opposite end was designed so that it could be sealed with replaceable rubber diaphragms. These allowed the flechettes to pass through while the integrity of the gas seal was maintained. Ports along the length of the cylinder allowed gas samples to be taken after firing. The apparatus is shown in Figure 2.

E. Gas Sample Flasks

Glass flasks (0.5 and 2l) with vacuum stopcocks, and tapered glass joints were used to sample the gas from the containment vessel. These were prepared by evacuation to ~ 5 mtorr, heating with a heat gun, and then filling with helium. This procedure was repeated several times before final evacuation. Checks by mass spectroscopy of the background in the sample flasks showed them to be free from contaminants.

F. Gas Condensation Train

The condensation train consisted of three gas traps in series, each of which could be isolated by vacuum stopcocks. They were immersed in cooling baths which in Run #1 were at 273, 195, and 77°K and in Run #2 were all at 77°K. After firing, a gas stream was pulled from the containment vessel through the traps with a vacuum pump.

G. Introduction of Gas Samples into GC/MS

A six-port gas valve with interchangeable sample loops was modified to allow injection of samples from the gas flasks into the GC. As depicted in Figure 3, the system was designed so that the sample loop could be evacuated with a vacuum pump to ~200 mtorr. The pump could then be valved off and an exhaust sample expanded into the sample loop from the gas flask.

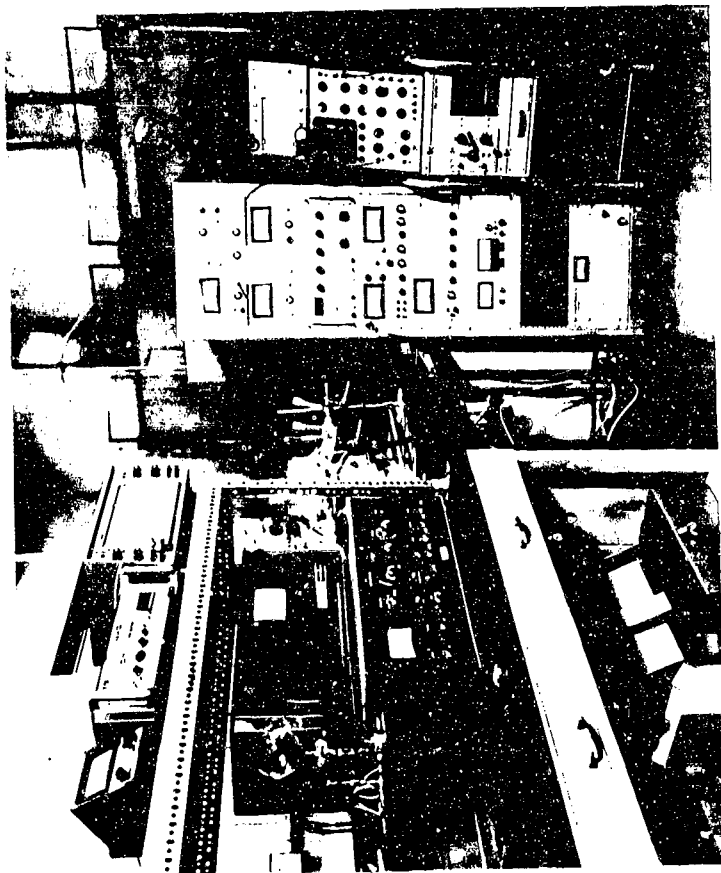


Figure 1. Gas Chromatograph/Mass Spectrometer System

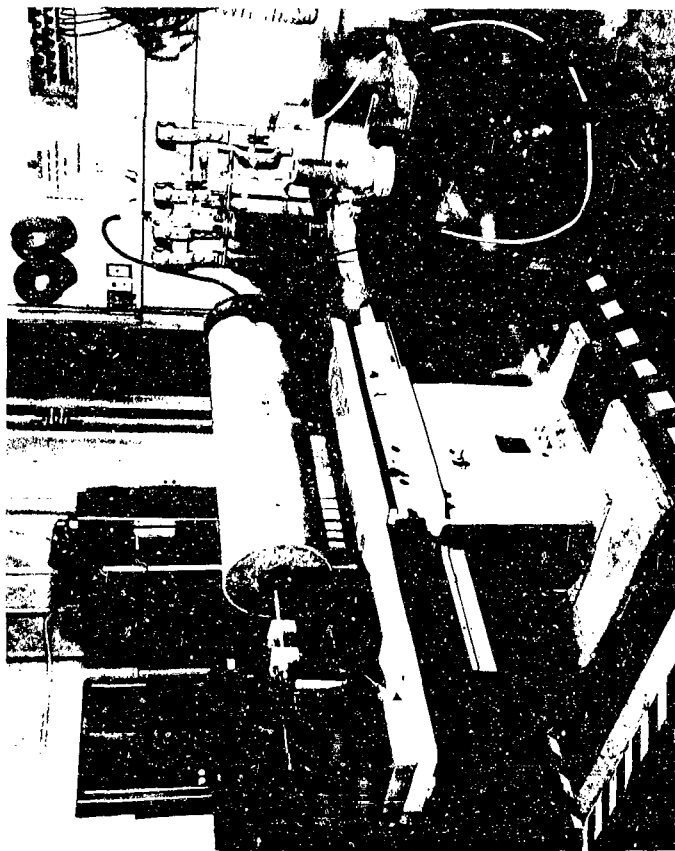
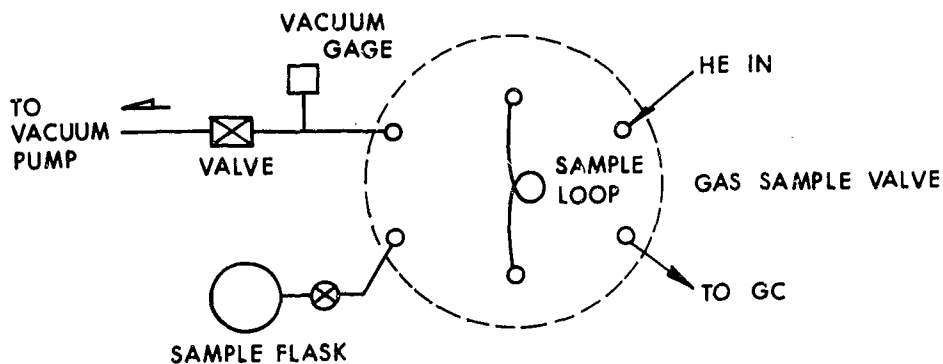
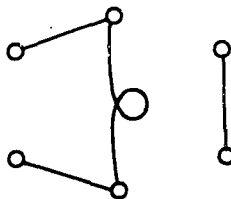


Figure 2. Exhaust Gas Sampling Apparatus . With XM-19 Rifle, Gas Containment Vessel and Cryogenic Gas Traps.



POSITION A:



POSITION B:

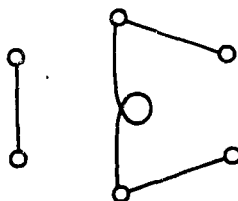


Figure 3. Schematic of Gas Sample Inlet System for Gas Chromatograph

Two different sample loops were used in this study, the first being a standard 5-ml loop. The second loop was designed to allow cryogenic concentration of dilute gas samples, and was constructed of a 30 cm length of 3-mm OD stainless steel tubing which was packed with Porapak QS. To concentrate a sample for injection, this loop was evacuated to ~200 mtorr and then immersed in liquid nitrogen. A gas sample was then pulled through the loop for 30 - 90 sec using the vacuum pump. After the pump and sample flask were valved off, the cooling bath was removed and the loop heated with a heat gun to ~70° before rotating the valve to inject the sample into the GC.

IV. EXHAUST SAMPLING PROCEDURE

Firings of the XM-19 were carried out in the Interior Ballistics Range of the Ballistic Research Laboratories following standard safety procedures. As shown in Figure 2 the rifle was mounted on a test bed with the muzzle within the gas confinement cylinder. Three-round bursts were fired by remote control. The flechettes passed through the rubber membrane on the cylinder and were captured in a sand-bag target down range. After the desired number of rounds had been fired, gas samples were taken by placing the tapered glass joint of a sample flask through one of the sample access ports in the cylinder. The stopcock was then opened and a sample of the exhaust/air mixture was drawn into the flask. When the static gas samples had been taken, the vacuum pump was turned on and gas drawn through the condensation train for five minutes. The individual traps were isolated and stored in their cooling bath until just before analysis when they were warmed and expanded into evacuated flasks.

Two firings of 12 rounds each were conducted to obtain samples for the qualitative and quantitative analysis of the exhaust gases. The containment vessel was purged with helium during the first firing to reduce the chance of post firing combustion of the gases in the vessel and to reduce the amount of air in the gas samples relative to the exhaust gases to aid in the qualitative analysis. In the second firing, the weapon exhaust was allowed to mix with the ambient air in the containment cylinder. No secondary explosions were observed during firing.

V. RESULTS

A. General

A preliminary investigation was necessary to develop the conditions for the gas chromatographic separations. A list of gases which might be found in the exhaust gas of the XM-19 was compiled and these are listed in Table I. A literature search was conducted to identify gas

TABLE I
RESULTS OF GC RETENTION TIME STUDIES ON POROUS POLYMER

COL ** Temp	COLUMN PACKINGS					
	P-QS 25°	P-QS 50°	P-QS 70°	P-S 25°	P-T 25°	P-QS+S 25°
GAS	Retention Time (sec)					
Air	33	30				72
CO	34					
NO	36					84
CH ₄	54		60	30		120 *
CO ₂	127	78				300
N ₂ O	174	105				390
C ₂ H ₂	246		120	204		695 *
C ₂ H ₄	246		120	150		610 *
C ₂ H ₆	378			204		910 *
H ₂ O		240				
H ₂ S	595	285				
CNCN		510				*
OCS		510				
HCN		600				
C ₃ H ₆			576			*
C ₃ H ₈			660			*

* Detectable by FID

** P-QS = Porapak QS

P-S = Porapak S

P-T = Porapak T

chromatographic column packings which would permit separation of these gases. The recently developed porous polymer packings appeared to have the most promise. These are commercially available as the "Porapak" series of gas chromatography packings².

A series of gas mixtures approximating the expected weapon exhaust was prepared and trial separations achieved on Porapaks QS, S, T and a series column of QS + S. The results of this study are listed in Table I. The most effective packing was Porapak QS which with proper temperature gave separation of all components except acetylene/ethylene and carbonyl sulfide/cyanogen. The former could be separated by coupling the Porapak S column to the rear of the QS column. The large increase in analysis time made this column arrangement undesirable. On this basis Porapak QS was chosen as the packing to be used in the exhaust analysis.

B. Qualitative Analysis

The TCD of the GC is sensitive to all compounds of interest in this study. The lower limit of detection for this device is ~20 ppm (with cryogenic enrichment trap). Collection and quantitative analysis of components present below this concentration was thus impossible with this detector.

The FID is sensitive to those compounds which have at least one oxidizable carbon atom. The lower limits for detection and quantitative analysis are below 1 ppm. Most of the permanent gases of interest in this study are not detected by this device. Table I indicates the components which are detected with the FID.

To facilitate the qualitative analysis of the exhaust, gas samples from the liquid nitrogen cooled condensation traps were subjected first to GC/MS analysis. These samples were enriched in those components whose boiling points are significantly above that of nitrogen (77°K). The gases with boiling points closer to nitrogen e.g. CO (81°K), CH₄ (111°K), NO (121°K) should show only slight enrichment.

The TCD and FID traces from typical chromatograms from Firings 1 and 2 are shown in Figures 4 and 5, respectively. The difference in sensitivities between these two detectors is evident. The components which were detected and identified in the exhaust on the basis of the mass spectra are listed in Table II. The number under each chromatographic peak in Figures 4 and 5 corresponds to the number of the component identified in Table II.

Mass chromatography involves the technique of monitoring the intensity of a single mass as a function of time during a gas chromatographic run. This technique is particularly useful in the search for a component which may be present in the effluent at very low levels.

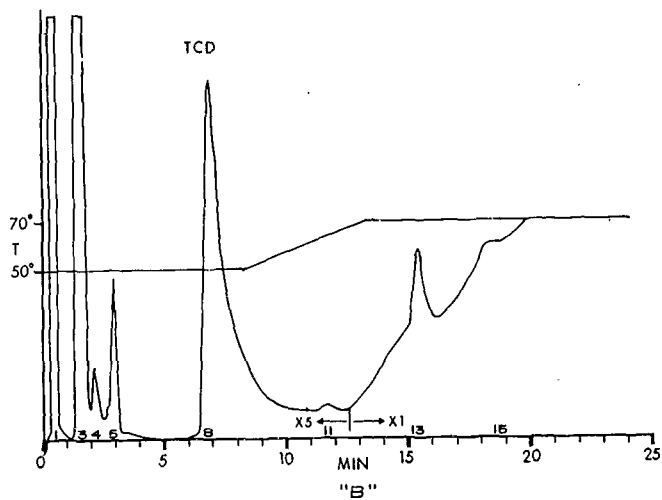
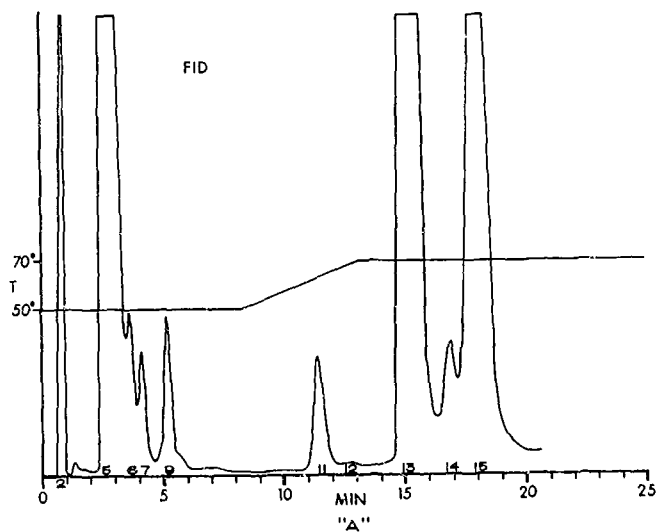


Figure 4. TCD and FID Traces from GC Analysis of Gases in Cryogenic Trap - Firing 1.

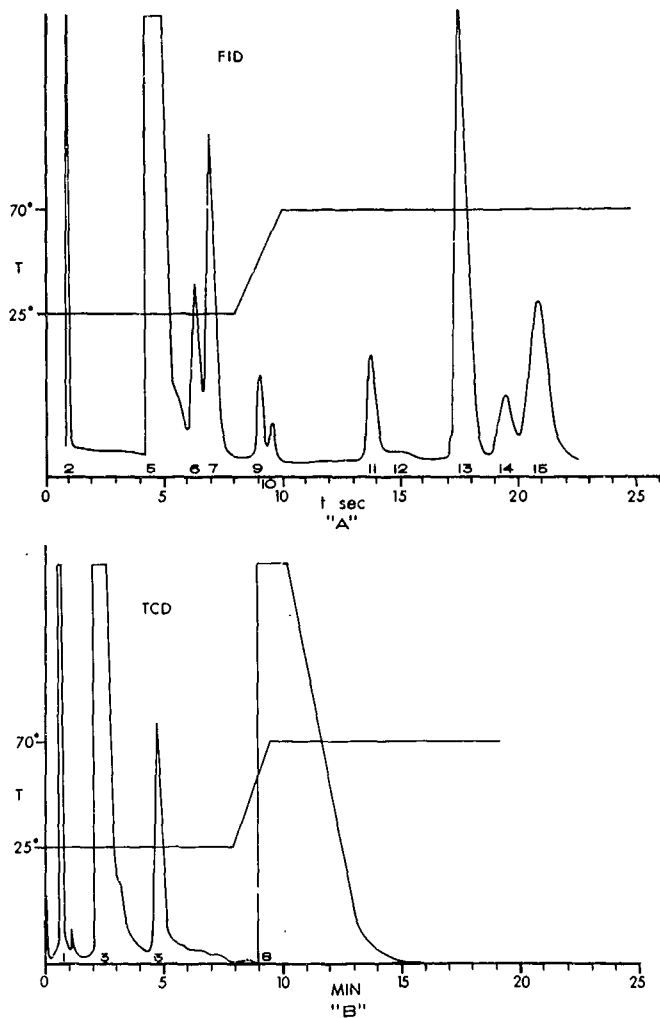


Figure 5. TCD and FID Traces from GC Analysis of Gases in Cryogenic Trap - Firing 2.

TABLE II
RESULTS OF QUALITATIVE ANALYSIS

Component (a)	Identity	Component (a)	Identity
<u>1</u>	Air	<u>7</u>	(b)
	CO	<u>8</u>	H ₂ O
	NO	<u>9</u>	(t)
<u>2</u>	CH ₄	<u>10</u>	(b)
<u>3</u>	CO ₂	<u>11</u>	CNCN
<u>4</u>	N ₂ O		OCS
<u>5</u>	C ₂ H ₂	<u>12</u>	HCN
	C ₂ H ₄	<u>13</u>	Propylene
<u>6</u>	C ₂ H ₆	<u>14</u>	Propane
		<u>15</u>	Propyne or Allene

(a) Numbers correspond to those in Figures 4, 5, 8.

(b) Identification could not be made due to small amount of component.

A prominent ion in its mass spectrum is observed and an increase in intensity of this ion at the known retention time of the component is good evidence for its presence in the mixture. This procedure can be followed for each prominent ion in the mass spectrum of the suspected component in order to obtain more evidence for its presence.

Figures 6 and 7 show a series of mass chromatograms obtained from the 70°K condensation trap of Firing 1. In Figure 6 the air peak gives a response at all M/e values 26, 28, 30, 34 due to N₂, O₂ molecular ions and their isotopic satellite peaks. CO₂ gives a response at M/e 28 (CO⁺) and 30 (C¹²O¹⁸+). The M/e 30 peak appearing after CO₂ is due to the (NO⁺) from N₂O whose GC retention time is slightly longer than that of CO₂ (cf. Table I). The large M/e 26 ion is due to C₂H₂⁺, the acetylene molecular ion.

Figure 7 depicts mass chromatograms for M/e 27, 34, 42, 60 ions along with the TCD trace from the gas chromatograph. It can be seen that the mass spectrometer gives a very large response to components which are not detected by the TCD or are buried in the noise. The M/e 60 ion is the molecular ion of carbonyl sulfide (OCS⁺), while the ion at M/e 34 can be attributed to the (S³⁴) fragment from this molecule. The molecular ion of propene (C₃H₆⁺) gives the M/e 42 peak while one of the principal fragments (C₂H₃⁺) results in the M/e 27 peak.

C. Quantitative Analysis

The quantitative analysis of the exhaust was complicated by the fact that some of the components (CO, NO, N₂O) were present at such low concentrations in the static gas samples that they were below the useable range of the TCD and are not detectable with the FID. Quantitative data for Firing 2 (conducted in ambient air) is available for those components which were present in sufficient concentration to be seen by the TCD or were detected by the FID. The concentration of CO was determined with an ultrasonic detector on a 3 m by 3 mm OD column packed with molecular sieve 5A. The results are listed in Table III. The concentrations of CO₂ and C₂H₂ in air were determined with the TCD. The ratios of the other components for which data are given were determined relative to C₂H₂ with the FID. These were then related to the concentration in air via the concentration of C₂H₂ determined by the TCD. Typical FID and TCD traces from the quantitative analysis of the static samples from Firing 2 are shown in Figure 8. The difference between the relative amounts of the various components in these traces and in those from the liquid nitrogen cooled condensation traps should be noted. The samples from the condensation traps show a large enrichment in the high boiling compounds (those which appear at longer retention times).

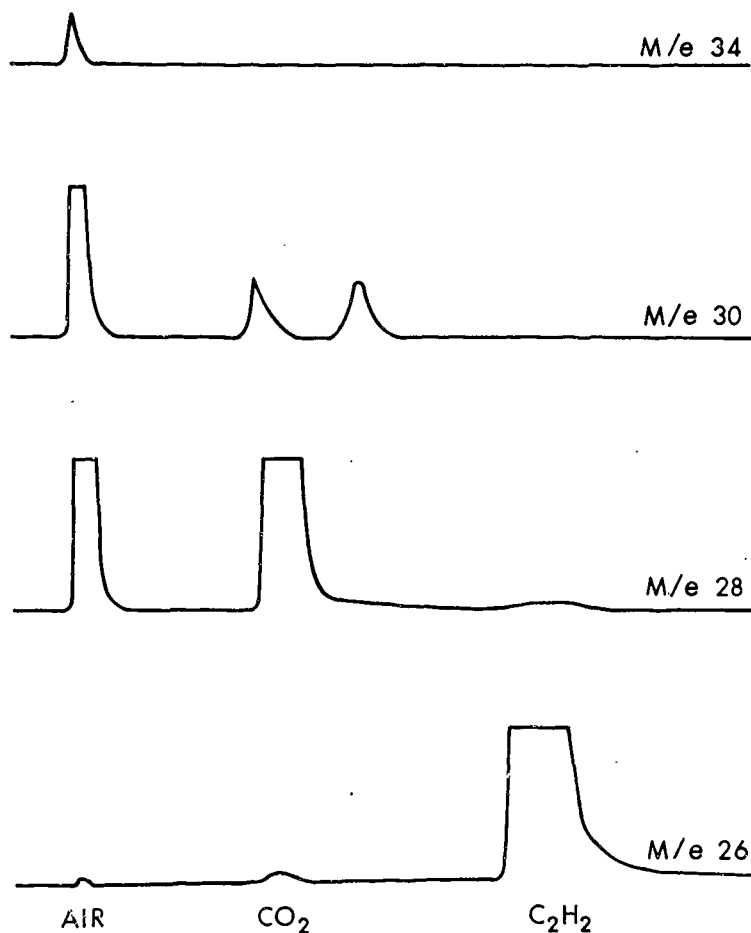


Figure 6. Mass Chromatogram: Ions at M/e 26,28,30,34 from Gases in Cryogenic Trap - Firing 1.

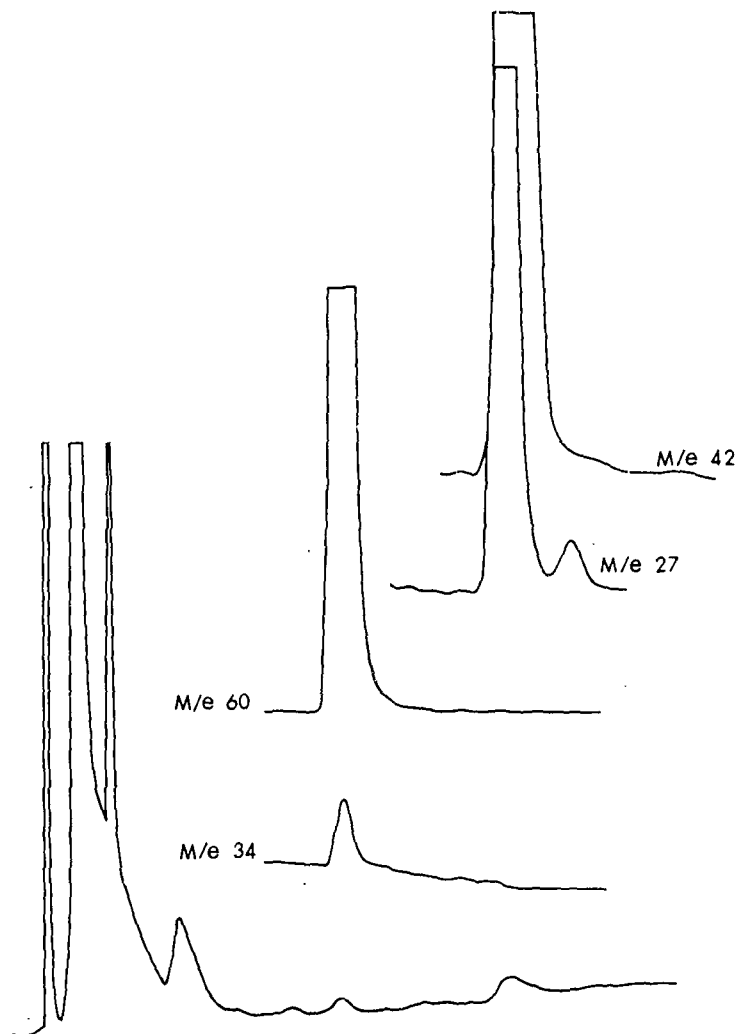


Figure 7. Mass Chromatogram: Ions at M/e 27,34,42,60 from Gases in Cryogenic Trap - Firing 1.

TABLE III
RESULTS OF QUANTITATIVE ANALYSIS

Component	Concentration (ppm) Relative to Air (2)	R ⁽¹⁾
CO	15,500	(1000)
CO ₂	5,500	380
CNCN	4	.25
OCS	4	.25
CH ₄	21	1
C ₂ H ₂ +C ₂ H ₄	19	1
C ₃ H ₆	15	1
C ₃ H ₈	<1	<.1
Allene or Propyne	<1	<.1

(1) $R = (\text{Concentration of component} / \text{concentration of CO}) \times 10^3$

(2) From 12 rounds in containment vessel

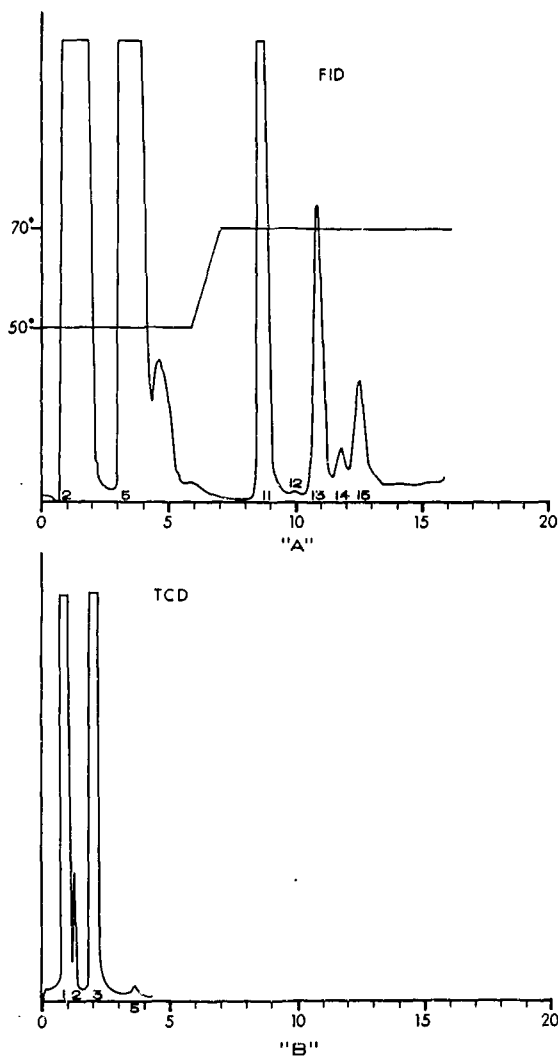


Figure 8. TCD and FID Traces from Quantitative GC Analysis of Ambient Gases in Gas Containment Cyclinder - Firing 2.

VI. THERMODYNAMIC CALCULATIONS

An attempt was made to calculate the final combustion products with an equilibrium thermodynamic code named "BLAKE". This code is a modification of the "TIGER" code developed by Stanford Research Institute under Ballistic Research Laboratories (BRL) sponsorship for detonation parameter calculations. (These modifications were made by Dr. E. Freedman, IBL, to whom all inquiries should be addressed). The modifications to this code now allow thermodynamic calculations of interest to gun interior ballisticians to be readily performed. Of primary interest in this study were, of course, the concentration results.

There are several comments to be made at this point. The library of elements was increased to include inorganic additives such as potassium sulfate. These ingredients are quite often ignored in thermodynamic equilibrium calculations since they have only minor effects on calculated performance parameters such as impetus, maximum pressure, and temperature. From a toxicological point of view, however, ingredients such as sulfur-containing compounds should be included. The products library was also increased to include more of the experimentally detected species. The library of final products is shown in Table IV. It should be mentioned, however, that this library is by no means complete, (NO_2 and N_2C_4 are omitted) and remains to some extent arbitrary.

In general, previous reports containing combustion exhaust calculations have quoted only the constant volume explosion results. It was felt that the normal gun combustion process, which is neither a constant volume nor a constant pressure process, might be more closely simulated by allowing the initial high temperature, high pressure state to expand and cool. The initial product concentrations calculated for a constant volume explosion are allowed to expand isentropically to an arbitrary final state. For lack of a better criterion the composition is arbitrarily considered frozen at the point at which the projectile exits from the muzzle and the hot combustion products expand and are cooled rapidly to ambient pressure. For the XM-19 rifle, this corresponds to an expansion ratio of approximately 10. This is a slightly different approach from that used by L. Stiefel in a recent Project West Report⁷. His calculations give best agreement with experiment if the equilibrium is frozen somewhere between 1000 and 5000 psi (6.9 and 34.5 MN/m²) for typical rifles which corresponds roughly to the muzzle pressure at projectile exit. The isentropic expansion assumption made in both cases neglects any heat loss and kinetic effects. Time dependent kinetic effects would result in composition changes lagging behind the assumed instantaneous equilibrium. Heat loss results in lower than calculated temperatures possibly aggravating any kinetic effects. It is impossible at the present time to predict accurately kinetic and heat loss effects on the chemistry involved.

TABLE IV
LIST OF CHEMICAL SPECIES INCLUDED IN
THERMODYNAMIC CALCULATIONS

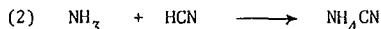
CO	GAS	S	GAS
H ₂ O	GAS	O ₂	GAS
H ₂	GAS	C ₂ H ₂	GAS
N ₂	GAS	C ₂ H ₄	GAS
CO ₂	GAS	CNCN	GAS
KOH	GAS	OH	GAS
H ₂ S	GAS	CN	GAS
NH ₃	GAS	HS	GAS
HCN	GAS	SO	GAS
K	GAS	CH ₃	GAS
CH ₂ O	GAS	H	GAS
COS	GAS	KO	GAS
NO	GAS	O	GAS
SO ₂	GAS	N	GAS
CH ₄	GAS	C	SOLID

Although the actual loading density of the XM-645 round in the chamber is approximately .95, this value was adjusted in the calculation to correspond with a more realistic maximum pressure. For the XM-645 a typical observed maximum pressure is approximately 45,000 psi (310.2 MN/m²). This corresponds to a constant volume explosion loading density of .25 gm/cm³ if the virial equation of state is used in the calculation. In essence the chamber is expanded by allowing the projectile to move to a point at which the new loading density will result in a reasonable pressure.

VII. DISCUSSION

The exhaust gas components identified on the basis of GC/MS data were (cf. Table II) CO, NO, CH₄, CO₂, N₂O, H₂O, OCS, CNCN, C₂H₂, C₂H₄, and propylene (C₃H₆). Some minor organic components detected by the FID (e.g. 6,7,9,10,12,14,15 in Figures 4,5) were not positively identified because of their low concentration or their elution closely following a major component which rendered the mass spectra difficult to interpret. Of these 6 is attributed to ethane on the basis of GC retention time and minimal MS data, and 14 to propane, also on the basis of retention time. Component 15 gave a mass spectrum which could be attributed to either methyl acetylene or allene. Component 12 had a retention time similar to HCN but corroborating MS data could not be obtained due to the low concentration. Component 5 was composed of a mixture of acetylene and ethylene with the former as the major component. No evidence was obtained for the presence of H₂S in the exhaust.

A trace of ammonia was detected in an infrared spectrum (1 m path length cell) taken immediately after sample collection. No evidence for NH₃ could be found in the GC/MS study. This can be explained by reactions in the gas mixture prior to GC/MS analysis. Typical reactions of this type would be (1) or (2).



NO was detected in low concentration in the GC/MS study while NO₂ was not detected. The low concentration of the former may be a result of its rapid oxidation by atmospheric oxygen to NO₂ at ambient temperatures. Analysis for NO₂ is at best very difficult due to its great reactivity (e.g. with GC column packing) and a tendency to be strongly adsorbed on surfaces (e.g. on the walls of the sampling system or GC columns), therefore, the presence of NO₂ cannot be ruled out. The inability to achieve good analysis for the oxides of nitrogen is unfortunate as the toxicity of these compounds exceeds that of CO.

The Threshold Limit Value (TLV), the maximum permissible concentration for 8 hr daily exposure in industry³, for CO is 50 ppm. Formation of these compounds is expected not only in the initial combustion reactions but also in those resulting in the secondary flash.

The quantitative analysis proved to be quite difficult. Several components (NO, N₂O, and OCS) could not be determined because their concentrations were well below the limit of the TCD and the FID does not respond to them. The concentration of OCS was estimated from the mass spectrometric data to be roughly equivalent to that of CNCN.

The major component of the exhaust was CO. Its concentration relative to air within the confinement vessel after firing 12 rounds is listed in Table III. The concentrations of the other components for which quantitative analyses were possible are also listed as a ratio of their concentration relative to that of CO.

The major components of toxicological interest are CO, OCS, and CNCN. The TLV values of NO, NO₂, and N₂O₃ are 25, 5 and ~ 8 respectively⁸. That for CNCN is 10 ppm while that for OCS has not been established but should be somewhat higher than that of H₂S (10 ppm)⁸. At a concentration of 100 ppm for 15 hr or 900 ppm for 1 hr, CO produces "severe distress" and becomes lethal at 4000 ppm (~ 1 hr)³. It can be concluded from the quantitative data that in a confined environment toxic levels of CO concentration should be reached long before the concentration of OCS or CNCN becomes significant. This conclusion is made somewhat tenuous by the lack of data comparing the relative toxicity of these compounds and effects of prolonged low level exposure.

Other gases such as ammonia and the oxides of nitrogen can produce irritation at concentrations below their toxic levels. Unfortunately quantitative data for these compounds were not available from the present study (*vide supra*).

It is possible that higher molecular weight compounds which might produce irritation may be present in the exhaust as an aerosol. Verification of this is beyond the scope and limitations of the present work.

A comparison of the calculated results normalized to carbon monoxide with experimental concentrations shows rather interesting trends (Table V). CO₂, COS, and CH₄ experimental values agree quite well with equilibrium results. (For details of the output see Appendix D).

A major puzzle is cyanogen, CNCN. The measured value is about five orders of magnitude higher than the calculated results. One explanation is the possibility that it is formed extraneous to the gun combustion process. A more likely explanation is that CNCN, a thermodynamically unstable molecule, may have a very high activation energy for any further possible reactions. Its concentration thus increases once it

TABLE V
COMPARISON OF EXPERIMENTAL AND CALCULATED PRODUCT
CONCENTRATIONS FOR X-2374.13 PROPELLANT*

	Calc.	Measured**		Calc.	Measured**
CO	1000	(1000)	C ₂ H ₂	2.86×10^{-5}	>1 ^b
H ₂ O	476	dnm ^a	C ₂ H ₄	3.80×10^{-5}	
H ₂	389		CNCN	1.57×10^{-8}	.25
N ₂	289	dnm	OH	5.97×10^{-4}	
CO ₂	364	380	CN	1.45×10^{-8}	
KOH	3.34		HS	3.73×10^{-3}	
H ₂ S	1.65		SO	8.27×10^{-5}	
NH ₃	3.66×10^{-1}	dnm	CH ₃	2.28×10^{-4}	
HCN	3.69×10^{-2}	dnm	H	1.01×10^{-2}	
K	1.97×10^{-1}		KO	8.43×10^{-7}	
CH ₂ O	1.98×10^{-2}		O	1.28×10^{-8}	
COS	1.18×10^{-1}	.25	N	3.10×10^{-10}	
NO	1.08×10^{-5}	dnm	C ₃ H ₄	(NI) ^c	<.1
SO ₂	3.35×10^{-4}		C ₃ H ₆	(NI)	.1
CH ₄	3.56×10^{-1}	1	C ₃ H ₈	(NI)	<.1
S	8.29×10^{-6}		C ₂ H ₆	(NI)	dnm
O ₂	7.27×10^{-9}	dnm			

* Values are normalized to CO. [(Concentration of component/
concentration of CO) x 10³]

** See Table III

a. Detected, but did not quantify.

b. Measured value includes both C₂H₂ and C₂H₄

c. Not included in these calculations

is formed and does not participate significantly in further equilibrium processes. This may very well be a typical example of kinetic lag. It is interesting to note that in Project West¹ essentially similar results were found. The calculations also indicate the presence of significant amounts of H_2S , HCN , SO_2 , and NH_3 , which were either not quantitatively identified or not detected. As is usual in most experimental investigations of this type a negative result does not necessarily indicate the absence of a constituent.

VIII. CONCLUSIONS

The results of this study agree with those of Project West¹. Carbon monoxide is the major volatile exhaust component of toxicological importance in a confined environment. Other toxic gases such as OCS and CNCN are present to less than 0.1% of the CO concentration. The lack of adequate data on the relative toxicity of these compounds does not permit them to be totally disregarded and work to provide this information is needed.

Gas chromatography/mass spectroscopy combined with cryogenic enrichment techniques has proved to be a powerful method for qualitative and quantitative analysis of complex gas mixtures. Further development of cryogenic enrichment techniques combined with a detector more sensitive to inorganic gases than the thermal conductivity detector should facilitate the quantitative analyses.

Mass chromatography has also proved to be a powerful technique which has the potential of providing both quantitative and qualitative data. The principal problem is one of data acquisition and reduction of large quantities of data. In addition, thermodynamic equilibrium calculations can be quite useful in the qualitative prediction of exhaust constituents. Quantitative predictions are, however, of somewhat more limited accuracy and discretion must be exercised in their use.

Future work should combine real-time analytical methods, such as those currently being developed for air pollution monitoring, with techniques such as those discussed here. Analyses should be conducted for higher molecular weight compounds which may be present in aerosols. Due to their high toxicity, further attention should be given to the analysis of the nitrogen oxides in weapons exhaust.

IX. RECOMMENDATIONS

Gun exhaust is toxic. Due care and consideration must be exercised by weapons system designers when guns are to be mounted in vehicles or aircraft. Proper exhaust arrangements must be provided in these cases.

ACKNOWLEDGMENTS

Many individuals provided their help and cooperation to make this study possible. Mr. George Samos, Dynamics Branch, IBL, BRL provided coordination with the U. S. Army Small Arms Systems Agency, and technical information throughout the project. Mr. Roger Bowman and SP4 Fred Schrieber assisted in the exhaust gas sampling experiments. The test firings were conducted using the facilities and with the help of the personnel of the Dynamics Branch, IBL, BRL. The contribution of the above is gratefully acknowledged.

We would also like to thank Dr. Ludwig Stiefel of Frankfurt Arsenal for his helpful comments and suggestions. Last but certainly not least we are grateful to Dr. Eli Freedman, Combustion and Propulsion Branch, IBL, BRL, who has been most helpful in the enlarging of the product library of "BLAKE", in its operation, and many fruitful discussions.

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GLOSSARY

GC	Gas Chromatography
MS	Mass Spectroscopy
TCD	Thermal Conductivity Detector
FID	Flame Ionization Detector
amu	Atomic Mass Units
CO	Carbon Monoxide
NO	Nitric Oxide
CH ₄	Methane
CO ₂	Carbon Dioxide
N ₂ O	Nitrous Oxide
NO ₂	Nitrogen Dioxide
H ₂ O	Water
CNCN	Cyanogen
OCS	Carbonyl Sulfide
HCN	Hydrogen Cyanide
H ₂ S	Hydrogen Sulfide
C ₂ H ₂	Acetylene
C ₂ H ₄	Ethylene
C ₂ H ₆	Ethane
C ₃ H ₆	Propylene
C ₃ H ₈	Propane
C ₃ H ₄	Propyne or Allene

APPENDIX A

A. Composition of X-2374.13 Propellant*

<u>Component</u>	<u>Wt. %</u>	<u>% of Total Wt.**</u>
Nitrocellulose	85.0	82.6
Nitroglycerine	9.4	9.1
Diphenylamine	0.9	.88
Dinitrotoluene	0.7	.68
Dibutylphthalate	2.8	2.7
Potassium Sulfate	0.5	.48
Moisture & Volatiles	0.7	.68
Weight = 1.3 grams		

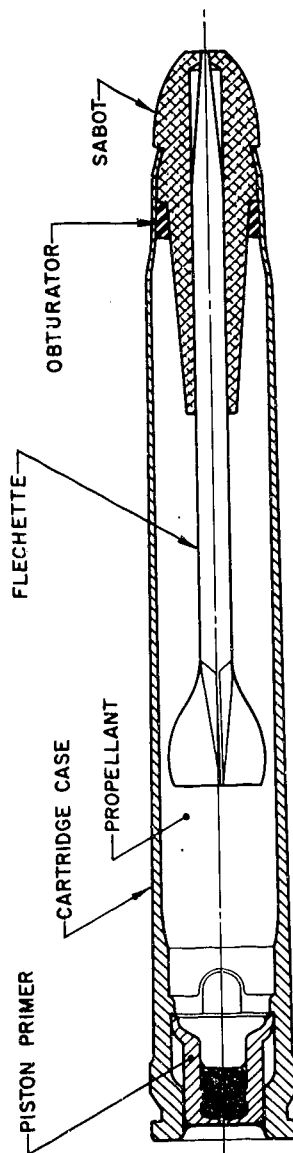
B. Composition of Piston Primer*

<u>Component</u>	<u>Wt. %</u>	<u>% of Total Wt.**</u>
Lead Styphnate	37 ± 5	1.02
Tetracene	4 ± 1	.11
Barium Nitrate	32 ± 5	.89
Antimony Sulfide	15 ± 2	.41
Aluminum Powder	7 ± 1	.19
PETN	5 ± 1	.14
Weight = 0.037 grams		2.8
Total**	1.337 grams	100%

* Nominal composition

** Total weight of propellant and primer

APPENDIX B



XM 645 Cartridge Assembly

APPENDIX C

Concentrations of Exhaust Gases From 7.62mm Machine Gun*

Component	Concentration (ppm)**	Concentration Relative To CO ($\times 10^3$)
CO	44,700	
CO ₂	9,800	220
NH ₂	90	2
HCN	90	2
CNCN	134	3
OCS	2	.04
CH ₄	670	15
C ₂ H ₂	90	2

* From reference 1

** From 100 rounds fired in N₂ atmosphere

APPENDIX D

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CONSTANT VOLUME EXPLOSION -NONIDEAL
SP14 R-2374-13
FEBRUARY 16, 1973
PAGE 1

THE COMPOSITION IS													
NAME	PERCENT BY WEIGHT	PERCENT BY MOLE	ENERGY OF FORMATION (CAL/MOLE)	FORMULA									
NC132	85.00	44.97	-0.1582200E 08	C	N	O	H						
				600	265	1031	735						
NG	9.40	6.17	-0.8376000E 07	C	H	O	N						
				300	500	900	300						
DPA	0.90	0.79	0.3148000E 07	C	H	N							
				1200	1100	100							
DNT	0.70	0.57	-0.1275000E 07	C	H	O	N						
				700	600	400	200						
DBT	2.80	1.50	-0.1906000E 08	C	H	O							
				1600	2200	400							
K2S04	0.50	42.74	-0.3415000E 07	K	S	O							
				200	600	400	200						
ETOH	0.50	1.62	-0.6413000E 07	C	H	O							
				200	600	200							
WATER	0.20	1.65	-0.6532400E 07	H	O								
				200	100								
THE ENERGY OF FORMATION FOR THE COMPOSITION IS -642.619 CAL/GRAM = -9571142.1 CAL/MOLE.													
COMPONENTS	CO2	H2O	N2	CO	CH4	KOH							
THE ELEMENTS AND PERCENT BY MOLE													
C	23.14												
H	37.45												
N	29.50												
O	29.50												
K	0.06												
S	0.03												
THERE ARE 29 GASEOUS CONSTITUENTS SELECTED WITH THEIR CONSTANTS													
NAME	BKN	CO-VOL	EPS/K	SIGMA	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)
1.	CO2	670.00	195.20	3.941	9.65915	-0.69946	0.12807	-0.00832	-3.27805	0.81665	-0.08553	-103112.7	59.8432
2.	H2O	360.00	809.10	2.541	6.50348	0.93789	-0.22670	0.01844	-3.80369	1.83571	-0.30223	-61992.6	47.8726
3.	N2	380.00	71.40	3.798	6.22733	-0.54937	0.00268	-0.00951	-2.73333	1.04067	-0.13919	-4895.8	50.9972
4.	CO	180.00	139.70	2.827	6.50771	-0.11572	0.02937	-0.01782	-0.13546	0.37822	-0.08873	-4597.7	37.5166
5.	CH4	180.00	139.70	2.827	6.50772	-0.11572	0.02937	-0.01782	-0.13546	0.37822	-0.08873	-4597.7	37.5166
6.	CO	390.00	91.70	3.477	6.11966	-0.54166	0.09643	-0.00635	-2.61463	0.83844	-0.10225	-31352.0	52.0925
7.	H2S	0.00	301.10	3.623	6.47063	0.00095	0.12112	-0.01788	-0.00199	-1.38583	0.41850	-13279.4	59.6567
8.	CH4	528.00	148.60	3.758	20.38251	-1.93871	0.26284	-0.01337	-14.43248	5.11197	-0.67906	-38010.8	38.5449
9.	KOH	0.00	150.00	3.000	7.27052	0.40176	-0.10705	0.00886	-1.70167	0.87842	-0.16491	-59699.7	68.1494
10.	KOH	159.00	344.70	3.339	7.48792	-0.25783	0.04424	-0.00222	-4.50416	1.73806	-0.25079	-24363.5	53.5559
11.	CSH4	372.00	224.70	4.163	22.63477	-1.64131	0.22022	-0.00917	-13.78191	4.49324	-0.59268	-10546.7	57.6777
12.	CSH4	372.00	224.70	4.163	22.63477	-1.64131	0.22022	-0.00917	-13.78191	4.49324	-0.59268	-10546.7	57.6777
13.	S2	0.00	100.00	3.000	7.61045	-0.16637	0.04532	-0.00323	-0.74195	-0.27124	0.07877	-78153.7	71.6890
14.	S	0.00	100.00	3.000	1.83331	0.19365	0.01065	-0.00358	0.77619	0.03824	0.02847	65713.8	47.5476

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15.	CH23	0.30	100.00	3.000	15.17766	-1.61928	3.25589	-0.01602	-8.72569	2.49889	-0.31366	-42755.0	53.6674
16.	N0	386.00	116.70	3.492	5.77838	-0.43972	0.18115	-0.00856	-9.78245	0.50895	-0.04564	16765.3	57.3869
17.	NH3	476.00	558.30	2.900	13.6392	-0.57028	0.18115	-0.00856	-9.78245	0.50895	-0.04564	16765.3	57.3869
18.	22	353.00	108.50	3.000	-2.32756	2.99499	-0.94677	0.12777	4.00797	-1.60009	0.24041	24101.5	45.8436
IV.	K	0.00	100.00	3.000	-2.32756	2.99499	-0.94677	0.12777	4.00797	-1.60009	0.24041	24101.5	45.8436

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THESE ARE 1 CONDENSED CONSTITUENTS SELECTED WITH THEIR CONSTANTS

NAME STATE(LIQUID/SOLIO) / STATE(LIQUID/SOLIO)

COMPONENTS	C ₀	H ₂ O	N ₂	KOH	H ₂ S
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ISOLINE (S.V.) AFTER CONSTANT VOLUME EXPLOSION SPIN X-2374.13

ISOLINE

LENNARD-JONES 6-12 EQUATION OF STATE

P (ATM)	V (CC/GM)	T (K)	H (CAL/GM)	E (CAL/GM)	S (CAL/K/GM)	RHO (GM/CC)	CV (CAL/K)	ALPHA	BETA	ADKXP	SIGMA (M/S)	OMEGA (K2/S2)
1) 3155.4963	4.0000	2934.	-336.94	-642.62	2.126	0.250	0.357	4.153	3.34	1.54	0.0	0.0
2) 1149.1537	8.0000	2418.	-600.08	-822.72	2.126	0.125	0.344	4.140	3.68	1.40	873.6	1.1
3) 449.9964	16.0000	2018.	-784.71	-959.08	2.126	0.062	0.338	4.114	3.87	1.32	1605.5	1.9
4) 182.9573	32.0000	1694.	-926.28	-1068.06	2.126	0.031	0.338	4.119	3.99	1.28	2246.3	2.5
5) 75.9662	64.0000	1431.	-1039.92	-1157.66	2.126	0.016	0.358	4.293	4.21	1.26	2820.3	2.9
6) 32.2142	128.0000	1230.	-1132.92	-1232.78	2.126	0.008	0.490	5.323	5.20	1.22	3339.4	3.3
7) 14.0896	256.0000	1098.	-1210.01	-1297.36	2.126	0.004	0.811	7.293	7.07	1.17	3812.3	3.7
8) 6.3275	512.0000	1009.	-1276.07	-1354.53	2.126	0.002	2.259	12.261	11.83	1.12	4250.3	3.9

CONSTITUENT CONCENTRATIONS - MOLES PER KGW OF EXPLOSIVE

NAME	1)	2)	3)	4)	5)	6)	7)	8)
CO GAS	1.76356E-01	1.75987E-01	1.68165E-01	1.61871E-01	1.53569E-01	1.42194E-01	1.27884E-01	1.12120E-01
H2O GAS	9.15833E-01	8.81790E-01	8.33478E-01	7.71030E-01	6.92244E-01	6.03769E-01	5.55762E-01	4.73608E-01
H2 GAS	4.31391E-01	5.18616E-01	5.67600E-01	6.29560E-01	7.03468E-01	7.68433E-01	7.75789E-01	7.65631E-01
N2 GAS	4.31391E-01	5.18616E-01	5.67600E-01	6.29560E-01	7.03468E-01	7.68433E-01	7.75789E-01	7.65631E-01
CO2 GAS	4.31391E-01	5.18616E-01	5.67600E-01	6.29560E-01	7.03468E-01	7.68433E-01	7.75789E-01	7.65631E-01
KOH GAS	5.31517E-02	5.31332E-02	5.35031E-02	5.44959E-02	5.59284E-02	5.57428E-02	5.61647E-02	5.63990E-02
H2S GAS	2.14738E-02	2.45197E-02	2.65113E-02	2.67081E-02	2.71351E-02	2.73632E-02	2.75179E-02	2.76262E-02
NH3 GAS	1.70551E-02	9.56930E-03	6.84235E-03	5.87209E-03	5.08912E-03	6.05655E-03	5.29449E-03	4.40244E-03
HCN GAS	9.42607E-03	3.11153E-03	1.28743E-03	5.98169E-04	3.10291E-04	1.63238E-04	7.55482E-05	3.48248E-05
CH4 GAS	5.26237E-03	1.73471E-03	6.96803E-04	3.22119E-04	1.57931E-04	7.95391E-05	1.48859E-05	1.26417E-05
COS GAS	3.12696E-03	2.69417E-03	2.34241E-03	1.91825E-03	1.57855E-03	1.32921E-03	1.18126E-03	1.06755E-03
N3 GAS	1.59960E-03	1.29430E-04	6.41990E-06	1.76116E-07	2.58030E-09	3.02940E-11	7.11840E-13	0.00000E-00
S2 GAS	1.59960E-03	2.32876E-04	4.57994E-05	5.42767E-06	3.22235E-07	2.47623E-08	2.85735E-09	6.32935E-10
CH4 GAS	1.58148E-03	1.17402E-03	1.96231E-03	5.78605E-03	2.79411E-02	1.55055E-01	8.82609E-01	8.12515E-01
C2H2 GAS	1.72685E-03	1.67161E-03	1.67684E-03	1.74261E-03	1.76761E-03	1.78761E-03	1.80761E-03	1.82761E-03
C2H4 GAS	2.75933E-06	4.50079E-06	1.22442E-06	6.43379E-07	2.23110E-07	1.13245E-07	5.63697E-08	1.30039E-08
C2H6 GAS	1.19552E-06	6.34154E-07	4.44031E-07	3.15857E-07	2.56611E-06	4.64437E-06	7.34912E-06	5.81512E-06
C2N2 GAS	4.57957E-07	2.47553E-08	2.23235E-09	2.55133E-10	3.27530E-11	4.36605E-12	5.76887E-13	7.44871E-14
OH GAS	1.54473E-02	2.08092E-02	1.82843E-04	9.57204E-06	2.98048E-07	7.44822E-09	3.26682E-10	2.60281E-11
CN GAS	7.95710E-06	3.00050E-07	9.98411E-09	2.55557E-10	3.51979E-12	5.05000E-00	0.00000E-00	0.00023E-00

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ISOLINE (S.V) AFTER CONSTANT VOLUME EXPLOSION SPIN X-2374.13

CONSTITUENT CONCENTRATIONS - MOLES PER KG OF EXPLOSIVE

NAME	1)	2)	3)	4)	5)	6)	7)	8)
HS GAS	2.22442E-03	9.05867E-04	2.73516E-04	6.05189E-05	9.80970E-05	1.22539E-06	2.88161E-07	8.49179E-08
SO GAS	5.37996E-04	1.24076E-04	1.69689E-05	1.33801E-06	1.33952E-06	4.82829E-07	1.64938E-07	2.37443E-11
CH3 GAS	2.08468E-04	4.12741E-05	1.12879E-05	1.44457E-04	1.42923E-05	1.06625E-06	1.51836E-07	4.31181E-08
H GAS	2.88018E-05	3.46951E-06	2.78669E-07	1.36533E-08	3.91148E-10	9.19266E-12	3.84259E-13	2.00526E-08
N GAS	6.03423E-05	1.97514E-06	3.12387E-08	2.08764E-10	5.59994E-13	0.00000E+00	0.00000E+00	0.00000E+00
W GAS	2.19676E-06	5.41604E-08	7.55127E-10	5.02446E-12	0.00000E+00	0.00000E+00	0.00000E+00	0.00000E+00
C(S)	0.00000E+00	0.00000E+00	0.00000E+00	0.00000E+00	0.00000E+00	0.00000E+00	0.00000E+00	1.95166E-21
TOTAL GAS (MOLES/KG)	40.8807	40.8757	40.8757	40.8691	40.8246	40.5701	39.9192	39.0603
DELTA Q (CAL/GM)	-768.180	-773.498	-778.758	-785.141	-794.153	-810.324	-837.221	-870.218

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1. ORIGINATING ACTIVITY (Corporate author) U. S. Army Ballistic Research Laboratories Aberdeen Proving Ground, MD 21005		2a. REPORT SECURITY CLASSIFICATION Unclassified	
		2b. GROUP	
3. REPORT TITLE ANALYSIS OF EXHAUST GASES FROM THE XM-19 RIFLE-- AN APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Memorandum Report			
5. AUTHOR(S) (First name, middle initial, last name) Joseph J. Rocchio and Ingo W. May			
6. REPORT DATE May 1973		7a. TOTAL NO. OF PAGES 50	7b. NO. OF REFS 8
8a. CONTRACT OR GRANT NO.		8b. ORIGINATOR'S REPORT NUMBER(S) BRL MEMORANDUM REPORT NO. 2293	
b. PROJECT NO. 1J63607D013			
c.		9b. OTHER REPORT ID(S) (Any other numbers that may be assigned this report)	
d.			
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13. ABSTRACT A technique combining gas chromatographic and mass spectrometric analysis was developed and applied to determine the chemical composition of gases resulting from firing the XM-19 rifle with the XM-645 flechette round. Cyanogen, carbonyl sulfide, carbon monoxide, nitrous and nitric oxides were among the products detected. Quantitative data were obtained for some of the components. Thermodynamic equilibrium calculations were performed and compared with the experimental results.			

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